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ON THE SIGNIFICANCE OF RADIATION TRAPPING IN THE INDUCTIVELY CO-ETC(U)

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TECHNICAL REPORT NO. 40

ON THE SIGNIFICANCE OF RADIATION TRAPPING  
IN THE INDUCTIVELY COUPLED PLASMA

by

M. W. Blades and G. M. Hieftje

Prepared for Publication

in

SPECTROCHIMICA ACTA, PART B

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Because of its proven analytical virtues, the inductively coupled plasma is now widely used. However, despite this proven utility, the development of ICP techniques is still in its infancy, hindered by a lack of understanding of excitation processes and the existence of unexplained interference effects. Because many of these interferences are themselves believed to involve excitation, a clearer understanding of excitation processes in the plasma is being sought by a number of workers.

It is now reasonably well established that excitation throughout the ICP is not accomplished by a completely thermal process (1-4). In particular, a number of studies (1-4) have shown that the analytical region of the plasma is not in complete local thermodynamic equilibrium (LTE). For example, Boumans and de Boer (5) have found emission intensities for some elements to be from one to three orders of magnitude greater than that predicted by LTE considerations.

Complicating any attempt to explain excitation in the ICP is the fact that the plasma is spatially inhomogeneous. Blades and Horlick (6) have found that LTE calculations could be applied successfully to the region from 0-15 mm above the load coil to predict spatial behavior of analyte atom emission; however, above this region the theory could not be applied.

The effect of concomitants on ICP emission underscores the importance of understanding better the excitation processes. For example, the effect of easily ionizable elements on analyte emission can be studied empirically (7) but even such detailed observations have not yet yielded a rational interpretation. Similarly, the addition of even small amounts of foreign gases (e.g. N<sub>2</sub>, He, or O<sub>2</sub>) can cause dramatic changes in the emission characteristics and spatial behavior of analyte species (8). This behavior

is almost certainly caused by changes in excitation conditions within the plasma gas.

Most excitation models which have been offered invoke argon metastable atoms as an important participant. A non-LTE mechanism for ionizing atoms and populating excited states in the ICP has been offered by Mermet (9) and is based on excitation transfer from metastable argon atoms. In this mechanism, metastable atoms play a role in excitation and ionization through a Penning process. Boumans and de Boer (5) offered a mathematical model whereby metastable argon acts as both a an ionizer and an ionizant and thus performs as a buffer species. On the surface, these explanations seem intuitively reasonable. The  $4^3P_0$  and  $4^3P_2$  states of argon ordinarily have long lifetimes because transitions between them and the ground state are forbidden. Because of this long lifetime, approximately 1.3 seconds (10), these states might serve as energy storage sites and thereby influence excitation in the ICP. Moreover, because these states are quite energetic (11.5-11.7 eV above the ground state), they would be capable of ionizing and/or exciting a large fraction of analyte species introduced into the plasma. Even the admixture of cold, sample-aerosol-containing argon to the hot plasma gases might then not diminish greatly the population density of the argon metastable species, thereby creating an argon metastable concentration far in excess of what would be predicted at temperatures in the analytical zone.

However reasonable the arguments involving metastable argon atoms, the foregoing models are incapable of explaining a number of observations. Although the metastable states are sufficiently energetic to excite many of the most sensitive analyte lines, a number of strong emission lines have

been observed which are above 11.7 eV. For example, Cd II (226.5 nm and 214.4 nm) and Zn II (202.5 nm) have higher energies. Alternative explanations must be proposed for excitation of these species, for example a two-step process involving ionization and subsequent excitation. Also arguing against a metastable-dominated excitation process is the likelihood that metastable atoms are readily converted to radiating species by collisions with other argon atoms (11, 12). The two metastable levels are intercalated by two radiative states  $4^3P_1$  and  $4^1P_1$ , the former 0.06 eV above the  $4^3P_2$  level and the latter 0.1 eV above the  $4^3P_0$  state. At typical plasma temperatures ( $10^4$  °K), the collision frequency ( $f_c$ ) between argon atoms at atmospheric pressure can be calculated from the following relationship.

$$f_c = \bar{v}/l$$

where  $\bar{v}$ , the rms velocity of the atom, is

$$\bar{v} = (3kT/m)^{1/2}$$

and  $l$ , the mean free path of the atom, is

$$l = [\sqrt{2} (n\pi d^2)]^{-1}$$

In equations (2) and (3),  $k$  is the Boltzmann constant,  $T$  is temperature in K,  $m$  is the atomic weight of the colliding atoms,  $n$  is their number density, and  $d$  their effective collision diameter. From these relationships, the collision frequency of metastable atoms would be approximately  $10^9$  per

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Eq. 1

Eq. 2

Eq. 3

second, suggesting the rapid interconversion of metastable to radiating states. Because the lifetimes of the radiating states are approximately  $10^{-6}$  sec (13), it would seem unlikely that either the metastable levels or their radiating counterparts would achieve an appreciable overpopulation compared to what would be predicted from the Boltzmann expression. Similarly, it would be surprising that such short-lived species could be carried intact from their point of production near the load coils to the analytical zone some 15-20 mm away.

Despite the foregoing reasoning, suprethermal excitation appears to exist in the ICP and metastable atoms have been measured (14, 15) in apparently high concentration in the analytical zone of the discharge. In this paper, we attempt to offer an explanation for these observations. In the resulting model, it will be argued that argon metastable species are indeed rapidly equilibrated with closely lying radiating levels. However, radiative deactivation which would ordinarily reduce the population of those levels is counteracted in large part by radiation trapping--the repetitive absorption and reemission of the excitation energy. Because of radiation trapping, the energy initially captured by the metastable states or by their radiative counterparts is retained within the plasma volume for periods as long as  $10^{-3}$  sec. As a result, Penning ionization and other processes in which metastable argon formerly figured significantly also involve radiative levels, not only those lying near the metastable states, but also those of higher energy. This model, it will be argued, can explain a number of hitherto poorly understood phenomena in the ICP and points toward the development of improved plasma sources.

### EXPERIMENTAL

Preliminary experiments were designed to probe the population densities of metastable argon atoms through use of absorption techniques. A number of other workers have employed similar procedures (14-16). Such measurements involve determining the absorbance at  $8115 \text{ \AA}$ , which corresponds to a transition from one of the metastable states ( $4^3P_2$ ) to a higher lying level (cf. Figure 1).

In the present study, these measurements were extended to a number of other transitions, which are also indicated in Figure 1. These other transitions correspond to absorption from not only the metastable levels, but from radiative levels as well. Upper levels involved in each transition corresponded in multiplicity to the ones from which the transition originated. To overcome any artifacts caused by temporal variations in either the primary source or the ICP itself, absorbances were measured simultaneously, using a photodiode array spectrometer. Conventional absorption geometry was employed throughout; a microwave-induced plasma (MIP) served as a source of argon line emission, which was focused on the ICP discharge and again onto a 0.35 m monochromator (GCA-McPherson Model 270). A Reticon 1024S photodiode array served as a detector and was interfaced for readout purposes to a MINC-11/03 computer. Consequently, lines over a  $500 \text{ \AA}$  range were measured simultaneously.

The MIP was operated at a forward power of 200 watts and supplied with argon gas at atmospheric pressure at a flow rate of 0.5 liters per minute. By means of a double stub tuner and microwave isolator, the MIP was tuned carefully and rendered sufficiently stable for the required observations.

To determine the absorbance from either metastable or radiating levels, three separate measurements were performed and involved the determination of, respectively, the MIP emission spectrum itself, the ICP background emission spectrum, and the combined MIP/ICP output, with the MIP radiation passing through the ICP discharge. By appropriate manipulation of these three signals, the absorbance of each of the lines detected by the photodiode array could be readily calculated. Figure 2 is an ICP emission spectrum of the region studied. The spectrum displayed in Figure 1 was determined at a height of 20 mm above the ICP load coils, at a forward power of 750 watts, a coolant gas flow rate of 15 liters per minute, a plasma gas flow of <1 liter per minute, and in the absence of nebulizer gas. Specific transitions corresponding to the lines in Figure 2 can be found in the diagram of Figure 1. Those lines marked with an \* correspond to those in which the lower level is a metastable state.

Absorbance values corresponding to the spectrum of Figure 2 are quantitated and tabulated in Table 1. Clearly, the  $8115^{\circ}\text{\AA}$  line is not a unique absorber. In fact, the absorbance from radiating levels are nearly as great as those from the metastable levels, suggesting comparable populations of the two kinds. Moreover, consideration of the oscillator strengths of the various transitions which are tabulated suggests strongly that radiating levels are populated to approximately the same extent as the metastable states.

### RESULTS AND DISCUSSION

From the absorbance (A) and oscillator strength (f) values collected in Table 1, the relative population densities of the closely lying metastable and radiating states near 11.5 eV can be calculated. Using these values and Equation 4, it can be shown that the population ratio of metastable ( $n_m$ ) to radiating ( $n_R$ ) levels is approximately 0.6.

$$\frac{n_m}{n_R} = \frac{A_m f_R}{A_R f_m} \quad \text{Eq. 4}$$

From this preliminary result, one must conclude that the metastable and radiating states are rapidly mixed through collisional processes. Consequently, the overpopulation of metastable levels which has been postulated (5) and measured (14,15) by others must also apply to the radiating states. It remains then to answer how the radiating levels ordinarily short-lived, do not immediately deactivate radiatively.

It is our thesis that the radiating levels, like metastable ones, serve to store energy through a phenomenon known as radiation trapping (17-19). It is known that noble gas transitions which couple to the ground state absorb strongly over a wide range of gas pressures (11, 18-19). Therefore, a quantum of resonance radiation emitted by one atom has a high probability of being reabsorbed by surrounding gas atoms, leading to a transfer of excitation from one atom to another. Obviously, at atmospheric pressure a large number of such emissions and reabsorptions can take place before radiation can eventually escape from the boundaries of the enclosed gas.

Hence the radiation is often considered to be "trapped". This radiation trapping leads to a longer apparent lifetime for the radiating levels and a consequent storage of the excitation energy in the enclosing gas.

Holstein (18, 19) treats this phenomenon quite clearly and suggests both the rapid conversion of metastable energy to radiating energy and the subsequent imprisonment of radiation. Conveniently, the apparent lifetime of an excited state which undergoes radiation trapping ( $\tau_{app}$ ) can be calculated from the expression (20)

$$\tau_{app} = \tau/\gamma \quad \text{Eq. 5}$$

where  $\tau$  is the natural lifetime of the transition and  $\gamma$  is a radiation imprisonment factor. For an infinite cylinder of the imprisoning gas having radius  $R$ ,

$$\gamma = \frac{1.6}{k_0 R (\pi \ln k_0 R)^{1/2}} \quad \text{Eq. 6}$$

where  $k_0$  is the absorption coefficient at the center of the transition given by

$$k_0 = \frac{\lambda^3 n g_2 A_{21}}{8\pi g_1 v_0 \sqrt{\pi}} \quad \text{Eq. 7}$$

where  $\lambda$  is the wavelength of the transition being imprisoned,  $n$  is the number density of excited states,  $A_{21}$  is the probability of the downward transition,  $g_1$  and  $g_2$  are the degeneracies of the lower and excited states in the transition, respectively, and  $v_0$  is the most probable velocity of atoms in the ground state.

For the 1048 Å (radiating to ground state;  $^3P_0 \rightarrow ^1S_0$ ) transition at  $10^4$  K in a conventional plasma of radius 0.8 cm, the ground state number density can be calculated to be approximately  $10^{18}$  cm $^{-3}$ . From these values, the apparent lifetime of radiating states would be  $1.16 \times 10^{-3}$  sec, far greater than the normal radiative lifetime of approximately  $10^{-8}$  sec. In an ICP torch where the linear argon flow is in the range of 30 m/sec, this extended lifetime would permit radiation to be trapped to a height of approximately 3 cm above the load coil, corresponding well to the actual height of the ICP emission plume.

The model suggested by radiation trapping can account for many observations in the ICP. In this model, metastable and radiating states would both be created in the same way that metastable levels were postulated to be. However, collisional equilibration between the two kinds of states would be rapid, but would result in little loss in excitation energy because of radiation trapping. Because such trapping would imprison the energy within the plasma volume for periods as long as 1 msec. sufficient time would exist for such processes as Penning ionization. Therefore, many of the existing models which invoke the presence of argon metastable species could be applied equally well to the new model, but with the provision that radiating levels be included.

However, a number of other ramifications of the new model exist. For example, Penning ionization need not arise only through a collision with excited argon species, but a similar process could also occur through interception of a photon imprisoned in the plasma volume. Similarly, the effect of foreign gases (e.g. O<sub>2</sub>, N<sub>2</sub>, etc.) could be explained by collision with excited argon species or by absorption of an imprisoned photon. Because the new model suggests high population of additional states and suggests the presence of im-

prisoned radiation, such ionization or deactivation processes should be rendered much more efficient, explaining even better the influence of foreign gases or the high excitation efficiency of the ICP.

The new model also provides an explanation for the unusually high excitation capability of the ICP. Although the predominantly trapped radiation would be that corresponding to the lowest radiating to ground state transition, other radiation trapping pathways exist. For example, the resonance transitions at  $879.9 \text{ \AA}$  and  $869.8 \text{ \AA}$  (cf. Figure 1) and  $876.1 \text{ \AA}$  and  $866.8 \text{ \AA}$  are also capable of trapping radiation. Moreover, although the probability of radiation trapping depends upon a substantial population in the lower level of the transition in question, it might exist for transitions between the lowest radiating or metastable states and higher ones (20-22), further contributing to the excitation of upper levels. As a result, one might expect that overpopulation of a number of excited levels would occur, leading to unusually high excitation energies that seem to be present in the plasma.

The new model also suggests new experimental directions to take with the ICP. Because radiation trapping is most efficient in a high-density medium, it would be most prevalent in high-pressure plasmas. In fact, operating an ICP at pressures above atmospheric might enhance radiation trapping and thereby contribute even more to the high excitation capability of the plasma. Conversely, at low pressures, radiation trapping would be minimized. However, at low pressures collisional equilibration between the metastable and radiating levels would be minimized and metastable excitation would become more important. Similarly, one would expect that larger plasmas would exhibit greater radiation trapping, because of their lower surface-to-volume ratio. Conversely, smaller plasmas might be less efficient in trapping radiation and thereby exhibit less excitation capability. This hypothesis might suggest a limit to how small ICPs can be made (23).

Obviously, the arguments presented in this paper must be considered somewhat speculative and are based on meager experimental evidence. However, the model we have offered agrees well with existing experimental evidence and previous (literature) findings. To prove or disprove the hypothesis, several additional experiments must be performed. First, it will be necessary to determine the time scale on which metastable and radiating states are equilibrated. Laser-based time-resolution experiments to determine this time scale are currently underway in our laboratory. In addition, it will be necessary to ascertain the presence of radiation trapping. Ordinarily, the existence of radiation trapping can be confirmed simply by monitoring the lifetime of the upper state involved in the transition. Unfortunately, the transition involved in the proposed model is in the far ultraviolet (1048 or 1067 Å) and cannot simply be examined. Other measurements to test the presence of radiation trapping might be those involving the influence of gas pressure in the ICP or the determination of the state lifetime through absolute absorption measurements. These experiments are also underway in our laboratory.

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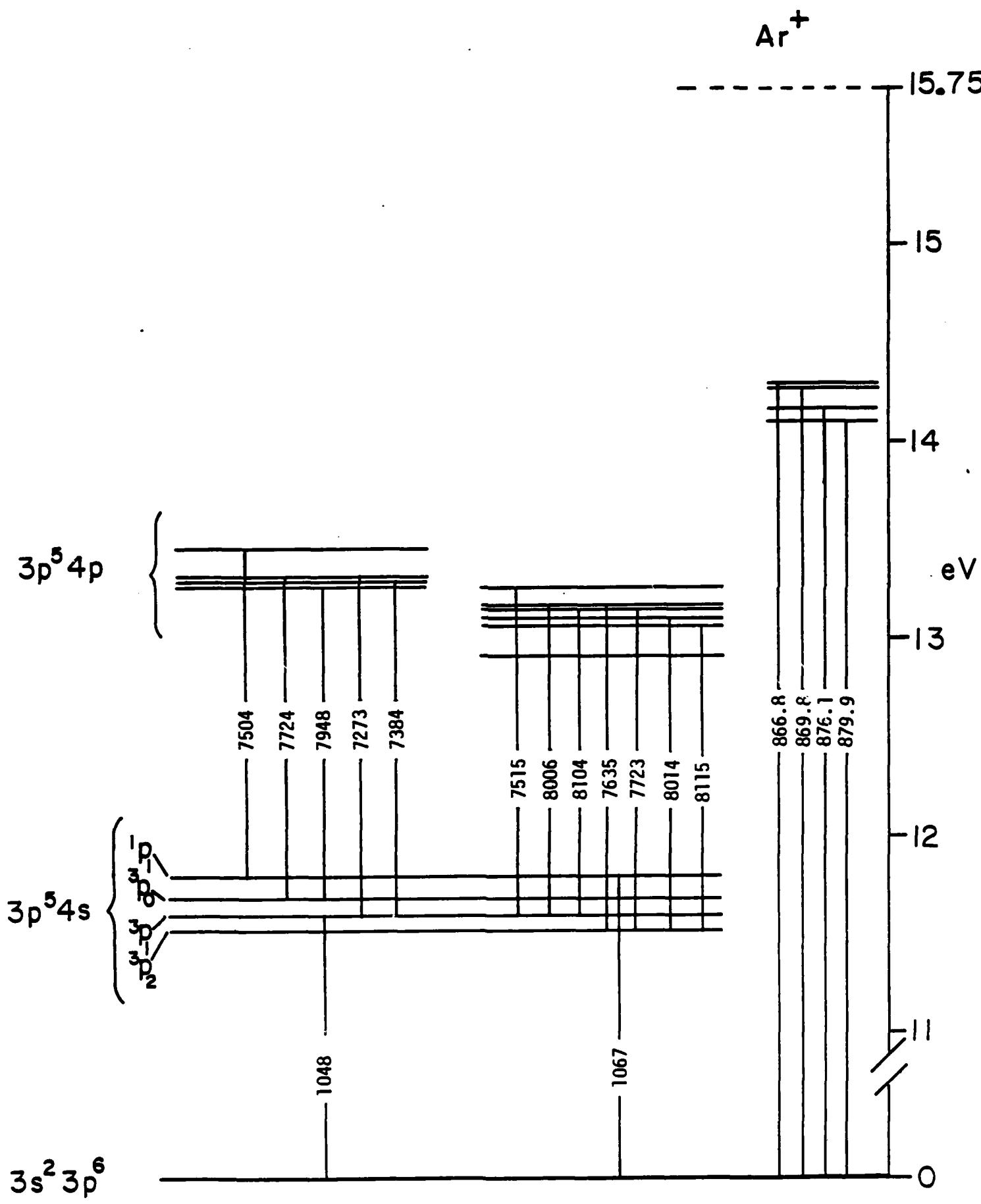
Table 1: Wavelengths, term symbols, absorbances, and oscillator strengths for lines used in absorbance measurements.

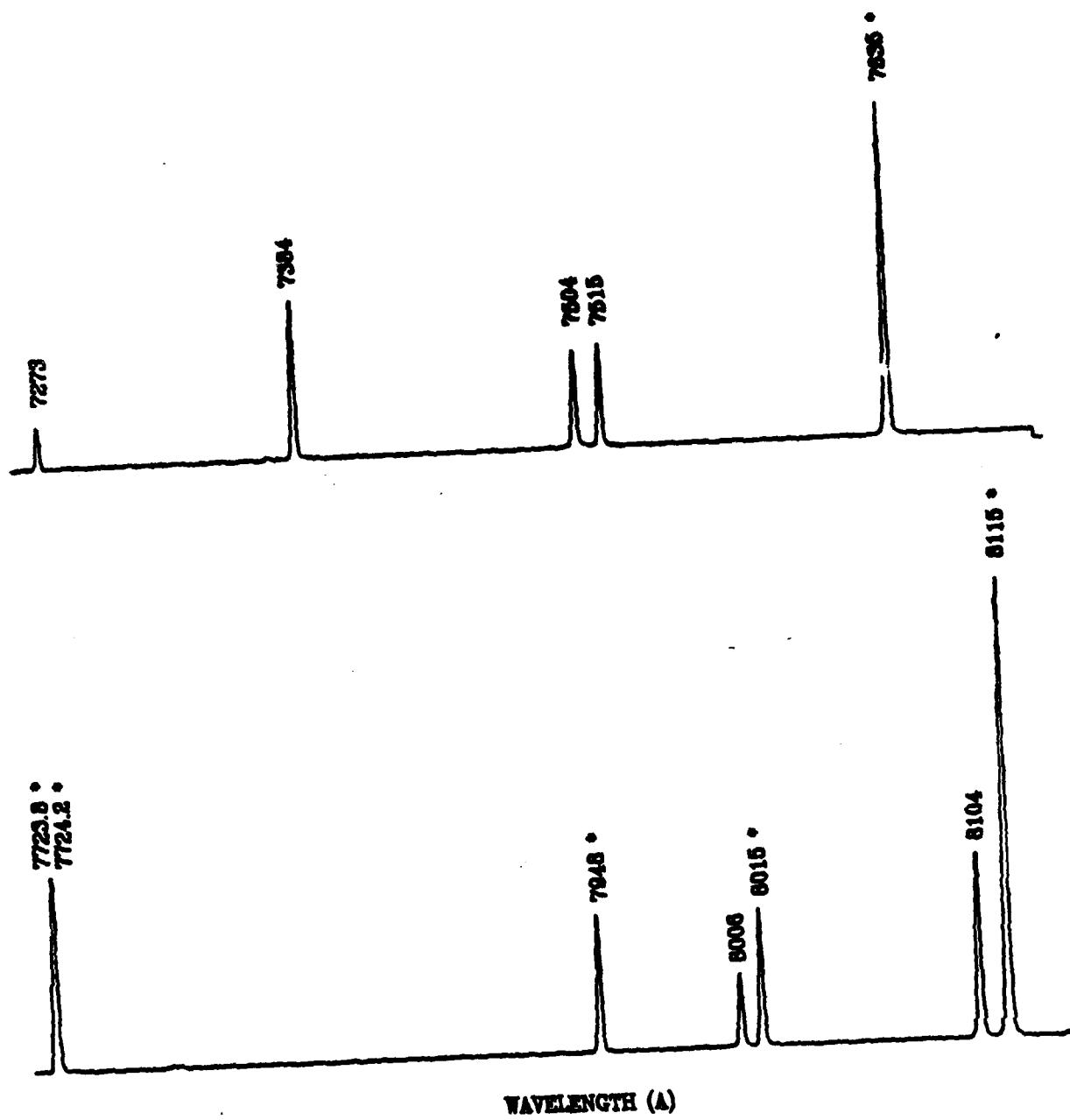
Wavelength	Transition	Absorbance	f
7273	4s[3/2] <sup>o</sup> - 4p'[1/2]	0.171	0.0159
7384	4s[3/2] <sup>o</sup> - 4p'[3/2]	0.116	0.119
7504	4s'[1/2] <sup>o</sup> - 4p'[1/2]	0.122	0.133
7515	4s[3/2] <sup>o</sup> - 4p'[1/2]	0.126	0.121
7635	4s[3/2] <sup>o</sup> - 4p[3/2]	0.146	0.239
7948	4s'[1/2] <sup>o</sup> - 4p'[3/2]	0.088	0.56
8006	4s[3/2] <sup>o</sup> - 4p[3/2]	0.108	0.075
8014	4s[3/2] <sup>o</sup> - 4p[3/2]	0.095	0.092
8104	4s[3/2] <sup>o</sup> - 4p[3/2]	0.091	0.273
8115	4s[3/2] <sup>o</sup> - 4p[5/2]	0.115	0.51

### Figure Legends

Figure 1. Partial term diagram for Ar I. Wavelength values in  $\text{\AA}^\circ$ .

Figure 2. Emission spectrum of ICP in the region  $7200 \text{ \AA}^\circ$  to  $8200 \text{ \AA}^\circ$ .  
Asterisks mark transitions whose lower level is metastable.





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